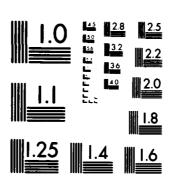
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Report 2346

DEVELOPMENT OF AN ACCELERATED CORROSION TEST FOR SCREENING ANTIFREEZE COMPOUNDS

by

James H. Conley and Robert G. Jamison

February 1982

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U.S. ARMY MOBILITY EQUIPMENT RESEARCH AND DEVELOPMENT COMMAND FORT BELVOIR, VIRGINIA

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The objective of this study was to develop an accelerated laboratory corrosion test for screening all antifreeze compounds, eliminating the need to conduct the costly and time-consuming simulated service test. The results of this phase of research indicate that this new accelerated bench corrosion test may replace the simulated service test.

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### **CONTENTS**

Section	Title	Page
	ILLUSTRATIONS	iv
	TABLES	iv
I	INTRODUCTION	1
11	APPARATUS	2
Ш	PROCEDURE	3
IV	RESULTS	6
v	CONCLUSIONS	10

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# **ILLUSTRATIONS**

Figure	Title	Page
1	Experimental Apparatus	3
2	Brass Bomb	4
3	Top with Electrode Assembly	5
4	Correlation Between Bench Corrosion Test and ASTM D-2570	9

# **TABLES**

Table	Title	Page
1	Accelerated Bench Corrosion Test Metal Coupon Weight Loss in Milligrams	7
2	Comparison of Accelerated Bench Corrosion Test with ASTM D-2570 Simulated Service Test	8
3	Regression Equations	8
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# DEVELOPMENT OF AN ACCELERATED CORROSION TEST FOR SCREENING ANTIFREEZE COMPOUNDS

#### I. INTRODUCTION

A three-phase program is necessary to determine the suitability of an antifreeze for actual service. This includes screening in glassware tests, testing in engine dynamometers or laboratory equipment capable of service simulation, and evaluation in vehicles on the road. The corrosion test in glassware is considered to be the first step in the evaluation of an antifreeze. It is a screening procedure for evaluating the effects of antifreeze solutions on metal specimens under controlled laboratory conditions. This method, ASTM D-1384, is generally capable of distinguishing between coolants that are definitely deficient from the corrosion standpoint and those that are worthy of further evaluation.

The second phase of the screening testing is normally conducted in the ASTM D-2570<sup>2</sup> Simulated Service Test. Simulated service testing offers improved and more selective coolant evaluation than is obtainable with glassware testing but is much more costly and time consuming. Features contributing to improved discrimination include the use of automotive cooling system components, a greater ratio of metal surface area to coolant volume and coolant circulation simulating that in a conventional automotive cooling system.

In the third and final phase the more rigorous full-scale engine and actual service tests are performed to obtain additional evidence of stability of coolant composition, inhibitor effectiveness, and service life.

Since many of the antifreeze compounds that successfully pass the first stage screening test do not pass the more costly simulated service test, an accelerated laboratory test that would reduce the number of simulated service tests would be a useful tool. This report describes the newly developed Accelerated Bench Corrosion Test and its application for testing antifreeze compounds.

<sup>&</sup>lt;sup>1</sup>ASTM D-1384 Corrosion Test for Engine Coolants in Glassware.

<sup>&</sup>lt;sup>2</sup>ASTM D-2570 Simulated Service Corrosion Testing of Engine Coolants.

#### II. APPARATUS

Figures 1, 2, and 3 describe the test apparatus. The apparatus consists of a brass bomb constructed according to the dimensions in Figure 2. A 600-ml lipless beaker is inserted into the brass bomb. Four hundred milliliters of a 33½-percent antifreeze solution diluted with ASTM corrosion water containing 100 p/m each of C1°, S0<sub>4</sub>, and C0<sub>3</sub> is used as the test solution. The sealed bomb is pressurized with 15 lb/in.² air and heated to 200°F while passing a 10 mA d.c. current through the electrode system. Figure 3 shows the position and arrangement of the anodic corrosion coupon assembly and the single cast iron cathode. The anode and cathode are insulated by use of teflon sleeves which also serve to maintain the 15-lb/in.² air pressure during the test.

#### III. PROCEDURE

#### Follow this procedure:

- 1. Assemble five cleaned and weighed corrosion coupons diagramed in Figure 3, using three No. 10 brass washers as a spacer group between each pair of metal coupons, and one brass washer at each end of the coupon assembly. Clean coupons according to instructions in ASTM D-2570 method and weigh to nearest milligram.
- 2. Clean and assemble cathode using the cast iron corrosion coupon weighed to nearest milligram.
- 3. Dilute antifreeze concentrate—1 part concentrate to 2 parts corrosion water (ASTM D-2570). Place 400 ml coolant and a 1-in. long magnetic stirring bar in the 600-ml-tall-form lipless beaker and place into the brass bomb.
- 4. Assemble "0" ring top, magnetic stirrer/heater or (preferably) a 500-W band heater, temperature probe and pressurize with 15 lb/in.<sup>2</sup> air.
- 5. Turn on stirrer and heater. Set stirrer at approximately 500 r/min. Allow fluid temperature to attain 200°F ± 5°F and then apply 10 mA d.c. current for 7½ h such that the cast iron electrode is cathodic (-), and the five-coupon assembly is anodic (±).

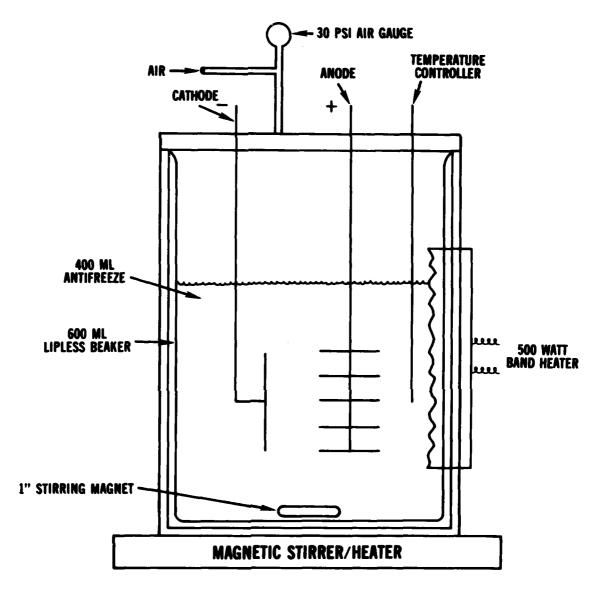


Figure 1. Experimental apparatus.

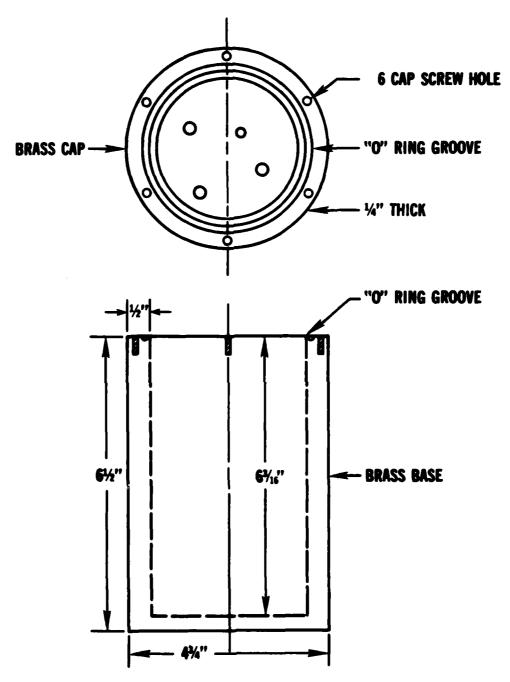


Figure 2. Brass bomb.

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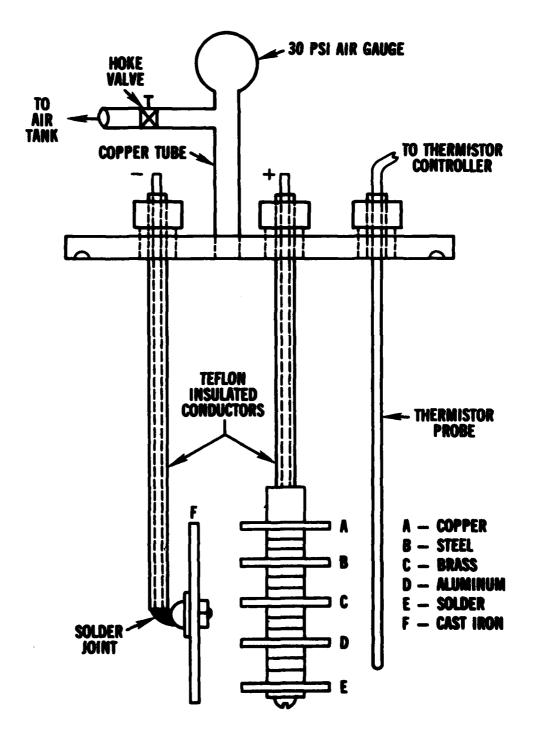


Figure 3. Top with electrode assembly.

- 6. Turn off current, heat, and air pressure and allow to cool overnight in sealed condition. Leave magnetic stirrer ON.
- 7. Next morning, turn on heat and air pressure. When temperature again reaches 200°F turn on 10 mA d.c. current for 7½ h.
- 8. Turn off current, heat, and air pressure and allow to cool to room temperature overnight in sealed condition.
- 9. Next morning, disassemble, clean, dry, and weigh corrosion coupons to nearest milligram.
  - 10. Compare coupon losses to allowable limits and report.

#### IV. RESULTS

Sixteen samples were tested in duplicate in the accelerated corrosion test apparatus. The samples included uninhibited ethylene glycol, MIL-A-46153 specification antifreeze, 7 commercial antifreezes, and 7 50/50 blends of MIL-A-46153 with the commercial antifreezes. The results tabulated in Table 1 show that half of the compounds gave high weight losses on the solder coupons. Those compounds should not be subjected routinely to further testing. Results of the uninhibited ethylene glycol gave high weight losses on both the steel and cast iron coupons.

Comparison of the accelerated test results versus the ASTM D-2570 test results are shown in Table 2. The results correlate except for Brand C which fails the accelerated test on the solder, copper, and brass coupons but fails on the aluminum and solder in the ASTM D-2570 test.

From the data in Table 2 for four of the antifreeze compounds the regression equations were calculated (Table 3) and the data are presented in Figure 4. It appears that in the case of Brands B and C there is a good linear correlation between the weight losses of similar coupons in the new accelerated bench test and the ASTM D-2570 method. A weak linear correlation was found for ethylene glycol and Brand A.

The preliminary results indicate that ethylene glycol, Brands B and G, and mixtures of Brand A with Brands B, C, D, E, and F should not be tested further. Brands A, C, D, E, F, and H mixtures of Brand A with Brands G and H could be considered for further testing.

Table 1. Accelerated Bench Corrosion Test Metal Coupon Weight Loss in Milligrams

Sample	Al	Solder	Cu	Br	Steel	C.I.
Ethylene Glycol	9	17*	23*	2	136*	25*
Brand A (MIL-A-46153)	36	9	1	2.5	1.6	0.34
Brand B	21.3	164*	6.6*	5	13*	3.2*
Brand C	29.2	18.9*	6.6*	7*	2	0.1
Brand D	1.4	23.5*	10.5*	6.8*	1.9	8.5*
Brand E	25.6	13.7	7.7*	5.4*	1.6	0.4
Brand F	38.5	24.4*	9.4*	8.5*	6.2*	0.2
Brand G	13	236.4*	9.6*	7.6*	7*	0.6
Brand H	34	10.5	0	5.7*	4.3*	1.0
50%A/50%B	2.7	456*	9*	10.8*	5.2*	0.2
50%A/50%C	5.9	185.9*	7.8*	6.6*	2.8	0.3
50%A/50%D	1.4	29.6*	7.5*	6.6*	1.9	0
50%A/50%E	+5.5	130.3*	8.5*	7.1*	2	0.4
50%A/50%F	24.7	95.5*	9.4*	9.8*	12.9*	0.85
50%A/50%G	14.4	15.2*	6.5*	5.5*	1.4	0.2
50%A/50%H	36.8	15.6*	8.3*	7.2*	1.4	0.65
Suggested Limits	40	15	5	5	3	1

<sup>\*</sup>Above suggested limits

Table 2. Comparison of Accelerated Bench Corrosion Test with ASTM D-2570 Simulated Service Test

<u> </u>		Acce	lerated E	ench T	est				ASTM	D-2570		
Sample	Al	So	Cu	Br	Steel	C.I.	Al	So	Cu	Br	Steel	C.I.
Ethylene Glycol	9	17*	23*	2	136*	25*	2	25	+0.2	0	113*	99+
Brand A (MIL-A-46153)	36	9	1	2.5	1.6	0.34	27	36	+0.2	+0.2	0.12	+6
Brand B	21.3	164*	6.6*	5	13*	3.2*	21.7	252*	1	4	+0.1	+1.2
Brand C	29.2	18.9*	6.6*	7*	2	0.1	365*	241*	6	5	5	5
Allowable Limits	40	15	5	5	3	1	120	60	20	20	40	40

<sup>\*</sup>Fails Limits

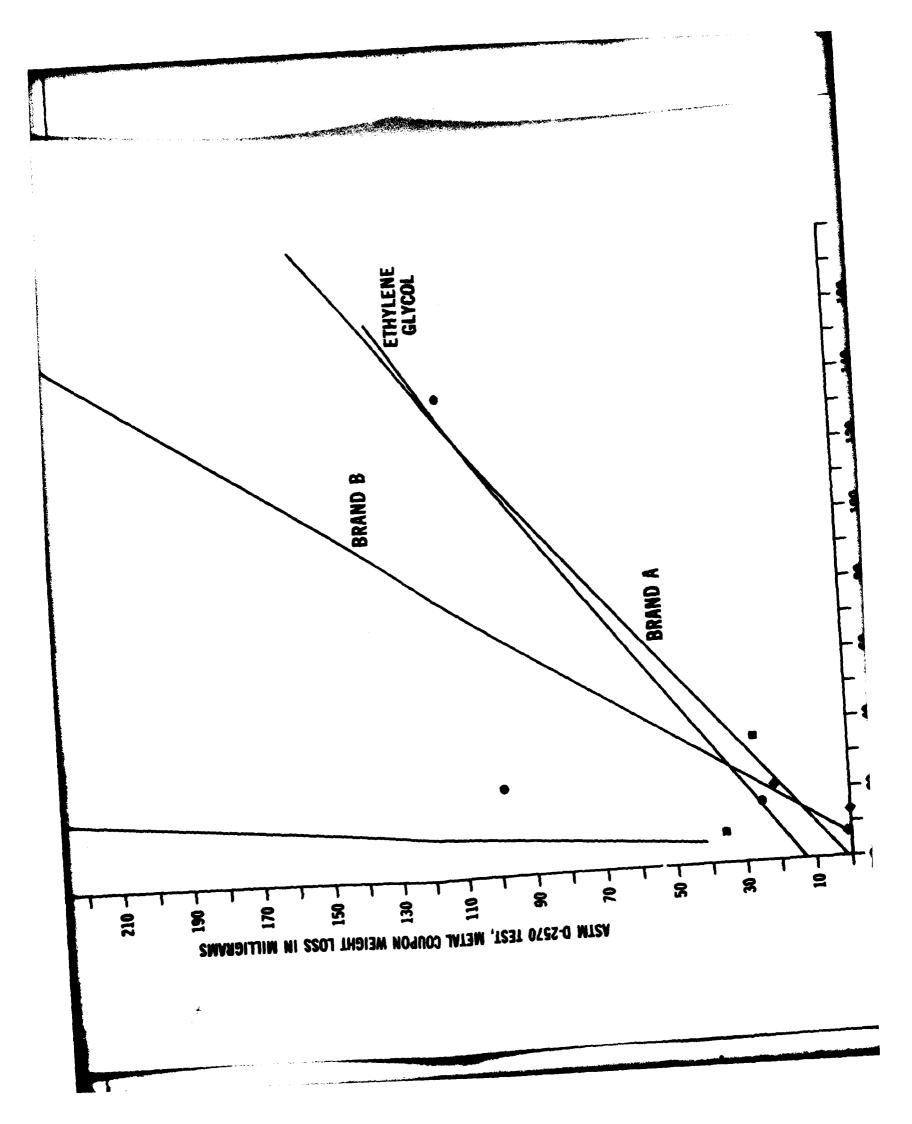
Table 3. Regression Equations

$- \gamma = 0.674$
y = 0.848 x + 2.34
$- \gamma = 0.998$
y = 1.596 x - 10.44
$- \gamma = 0.971$
$y = 13.74 \times -41.64$
0.7514
$- \gamma = 0.7514$ $y = 0.785 x + 12.05$

y = ASTM D 2570

x = Accelerated Bench Test





#### **V. CONCLUSIONS**

Indications are that one half of the samples tested should be eliminated from further testing based on the fact that the accelerated test gives a good indication of solder corrosion, which is one of the most serious offenders in today's cooling systems. Determination of reproducibility and repeatability of the new method and its correlation with the ASTM D-2570 method will require additional work.

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